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REMARKS

This responds to the Office Action mailed on May 17, 2004.

Claims 36, 37, 38, 59, 71, 74, 77, and 80 are amended, no claims are canceled, and no claims are added; as a result, claims 36-39, 59-61, 71-85, and 98-99 are now pending in this application. The amendments to the claims are fully supported by the specification as originally filed. No new matter is introduced. Applicant respectfully requests reconsideration of the above-identified application in view of the amendments above and the remarks that follow.

Support for claim 36 is found in the specification, for example, on page 8, lines 20.

Support for claim 37 is found in the specification, for example, on page 8, lines 21-25.

Support for claim 38 is found in the specification, for example, on page 13, line 18.

Support for claim 59 is found in the specification, for example, on page 15, lines 24-25.

Support for claims 71, 74, 77, and 80 is found in the specification, for example, on page 13, line 26- page 14, line 1, page 14, lines 21-29, and Figure 4.

§102 Rejection of the Claims

Claim 37 was rejected under 35 USC § 102(e) as being anticipated by Weitzel et al.(U.S. 5,661,312). Applicant traverses these grounds of rejection of this claim.

Applicant reserves the right to swear behind Weitzel et al (hereafter Weitzel) at a later date. Nevertheless, Applicant submits that claim 37 is distinguished over Weitzel.

Applicant cannot find in Weitzel a disclosure, a teaching, or a suggestion of a gate formed of $Si_{1-x}C_x$ with x between 0.6 and 1.0 as recited in claim 37, as amended. Therefore, Weitzel does not anticipate claim 37, and claim 37 is patentable over Weitzel.

Applicant respectfully requests withdrawal of these rejections of claim 37, and reconsideration and allowance of this claim.

§103 Rejection of the Claims

Claims 36 and 38-39 were rejected under 35 USC § 103(a) as being unpatentable over Chiang et al. (U.S. 4,598,305) in view of Halvis et al. (U.S. 5,369,040).

Claims 59-61 were rejected under 35 USC § 103(a) as being unpatentable over Chiang et al. and Halvis et al.

Claims 37 and 99 were rejected under 35 USC § 103(a) as being unpatentable over Chiang et al. in view of Halvis et al.

Applicant traverses these grounds of rejection of these claims. Applicant cannot find in the combination of Chiang et al. (hereafter Chiang) and Halvis et al. (hereafter Halvis) a teaching or a suggestion of a gate formed of $Si_{1-x}C_x$ where x is less than 1.0 and substantially greater than 0.5 as recited in claim 36, as amended, where x is between 0.75 and 1.0 as recited in claim 59, as amended, and where x is between 0.6 and 1.0 as recited in claim 37, as amended. Chiang deals with a photodetector having a source, drain, and gate, but as stated in the Office Action, Chiang does not teach or suggest a $Si_{1-x}C_x$ gate. The Office Action applies Halvis to disclose a polysilicon gate with up to 50% carbon, which is a distinctly different $Si_{1-x}C_x$ composition than is recited in claims 36, 37, or 59. Hence, the combination of Chiang and Halvis does not teach or suggest all the elements of claims 36, 37, or 59. Further, with Halvis disclosing a polysilicon gate with up to 50% carbon, the combination of Chiang and Halvis teaches away from the $Si_{1-x}C_x$ composition recited in claims 36, 37, and 59 as amended. Thus, Applicant submits that claims 36, 37, and 59 are patentable over Chiang in view of Halvis.

With respect to claim 61 and 99, the Office Action states “there is no difference in the meaning of the verbiage ‘nanocrystalline’ and the meaning of the verbiage ‘amorphous’ because even in an ultra-amorphous state nanocrystals, i.e., crystals at the scale of the interatomic distance, exist. Therefore, the list of crystalline forms that constitutes the essence of this claim is not a true limitation.” With respect to these instant claims, Application respectfully disagrees. The Office Action has not provided a reference or support that nanocrystalline is limited at the scale of an interatomic distance. “Nano” in nanocrystalline deals with scale on the order of nanometers, which may include several nanometers. Applicant includes with this response a copy of an article from a previously reviewed Information Disclosure Statement filed 28 July 2003 with respect to the instant application that provides a non-limiting example of the term “nanocrystalline.”

Claims 38 and 39, claim 99, and claims 60 and 61 depend on claims 36, 37, and 59, respectively, and are patentable over Chiang in view of Halvis for at least the reasons stated above, and further in view of the elements of these dependent claims.

Applicant respectfully requests withdrawal of these rejections of claims 36-39, 59-61, and 99, and reconsideration and allowance of these claims.

§103 Rejection of the Claims

Claims 36 and 98 were rejected under 35 USC § 103(a) as being unpatentable over Nakamura et al. in view of Halvis et al.

Applicant traverses these grounds of rejection of these claims. Applicant cannot find in the combination of Nakamura et al (hereafter Nakamura) and Halvis a teaching or a suggestion of a gate formed of $Si_{1-x}C_x$ with x less than 1.0 and substantially greater than 0.5 as recited in claim 36, as amended. Nakamura deals with a device structure which the authors refer to as a floating gate pixel, which “consists of a coupling capacitor, a photogate, a barrier gate, and a MOS transistor.” Nakamura has a floating gate node connected to the coupling capacitor, to the photogate, and to the gate of the MOS transistor. However, Nakamura does not teach or suggest a $Si_{1-x}C_x$ gate. The Office Action combines Nakamura with Havel since, as stated in the Office Action, Havel’s gate “can be made more transparent by adding up to 50% carbon.” It is apparent that the Office Action applies Havel’s gate having transparency properties to Nakamura as Nakamura’s photogate to disclose a polysilicon gate with up to 50% carbon, which is a distinctly different $Si_{1-x}C_x$ composition than is recited in claim 36. Hence, the combination of Nakamura and Halvis does not teach or suggest all the elements of claim 36. Further, with Halvis disclosing a polysilicon gate with up to 50% carbon, the combination of Nakamura and Halvis teaches away from the $Si_{1-x}C_x$ composition recited in claim 36 as amended. Thus, Applicant submits that claim 36 is patentable over Nakamura in view of Halvis. Claim 98 is dependent on claim 36 and is patentable over Nakamura in view of Halvis for at least the reasons stated above and further in view of the elements of dependent claim 98.

Applicant respectfully requests withdrawal of these rejections of claims 39 and 98, and reconsideration and allowance of these claims.

§103 Rejection of the Claims

Claims 71-73 were rejected under 35 USC § 103(a) as being unpatentable over Nakamura et al. (ISSN #: 0018-9383) in view of Halvis et al.

Claims 74 and 76 were rejected under 35 USC § 103(a) as being unpatentable over Nakamura et al. in view of Halvis et al.

Claims 77 and 79 were rejected under 35 USC § 103(a) as being unpatentable over Nakamura et al. in view of Halvis et al.

Claims 80-82 were rejected under 35 USC § 103(a) as being unpatentable over Nakamura et al. in view of Halvis et al.

Claim 75 was rejected under 35 USC § 103(a) as being unpatentable over Nakamura et al. and Halvis et al. as applied to claim 74, and further in view of Chiang et al.

Claim 78 was rejected under 35 USC § 103(a) as being unpatentable over Nakamura et al. and Halvis et al. as applied to claim 77, and further in view of Chiang et al.

Applicant respectfully traverses these rejections of claims 71-82, Applicant submits that the proposed combination of Nakamura and Halvis by the Office Action does not teach or suggest the elements as recited in the independent claims 71, 74, 77, and 80, as amended. The primary reference cited by the Office Action is the article by Nakamura entitled “CMOS Active Pixel Image Sensor with Simple Floating Gate Pixels.” Nakamura deals with a device structure which the authors refer to as a floating gate pixel. Nakamura’s floating gate pixel “consists of a coupling capacitor, a photogate, a barrier gate, and a MOS transistor.” On the other hand, claims 71-82 deal with a floating gate transistor in which a control gate is disposed on a floating gate, where the floating gate comprises $Si_{1-x}C_x$.

Applicant cannot find a teaching or a suggestion in Nakamura of a floating gate transistor where a control gate is on and separated from a floating gate by an intergate dielectric as recited in the amended independent claims 71, 74, 77, and 80. Nakamura has a floating gate node connected to the coupling capacitor, to the photogate, and to the gate of the MOS transistor. Further, Nakamura does not teach or suggest a $Si_{1-x}C_x$ gate. The Office Action combines Nakamura with Havel since, as stated in the Office Action, Havel’s gate “can be made more transparent by adding up to 50% carbon.” It is apparent that the Office Action applies Havel’s gate having transparency properties to Nakamura as Nakamura’s photogate. There does not appear to be a motivation to use Havel’s gate in the MOS transistor of Nakamura. Therefore, the combination of Nakamura and Havel does not teach or suggests all the elements as recited in independent claims 71, 74, 77, and 80. Thus, Applicant submits that the independent claims 71,

74, 77, and 80 are patentable over Nakamura in view of Halvis for at least the reasons stated above. Additionally, the claims that depend from independent claims 71, 74, 77, and 80 are patentable over Nakamura in view of Halvis for at least the reasons stated above and in further view of the elements of these dependent claims.

Applicant cannot find in Chiang a teaching or suggestion of a floating gate as recited in the independent claims 74 or 77. Therefore, Chiang does not cure the abovementioned deficiencies of applying the combination of Nakamura and Halvis to these independent claims. Thus, the combination of Nakamura, Halvis, and Chiang does not teach or suggest all the elements of the independent claims 74 and 77. Applicant submits that the independent claims 74 and 77 are patentable over Nakamura in view of Halvis in further view of Chiang. Further claims 75 and 78 depend from independent claims 74 and 77, respectively, and are patentable over Nakamura in view of Halvis in further view of Chiang for at least the reasons stated above and in further view of the elements of these dependent claims.

Applicant respectfully requests withdrawal of these rejections of claims 71-82, and reconsideration and allowance of these claims.

Allowable Subject Matter

Claims 83-85 were indicated to be allowable over the prior art.

Applicant acknowledges allowance of claims 83-85.

CONCLUSION

Applicant respectfully submits that the claims are in condition for allowance and notification to that effect is earnestly requested. The Examiner is invited to telephone Applicant's attorney at (612) 371-2157 to facilitate prosecution of this application.

If necessary, please charge any additional fees or credit overpayment to Deposit Account No. 19-0743.

Respectfully submitted,

LEONARD FORBES ET AL.

By their Representatives,

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Date 19 July 2004

By David R. Cochran
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Reg. No. 46,632

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Signature

Amy

Nanocrystalline Si: a material constructed by Si quantum dots.

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Abstract

Intense blue luminescence with decay lifetimes of 100–500 ps is observed from nanocrystalline silicon thin films at room temperature. The grain size reduction of the silicon crystallites to 3–5 nm leads to the generation of the luminescence. It is demonstrated that the blue emission is due to direct transitions in silicon nanocrystallites which are caused by a quantum confinement effect. The contribution of silicon dioxide to the blue emission is excluded. A model is proposed to understand the enhancement of the direct transitions in silicon nanocrystallites.

Keywords: Silicon; Quantum structures; Quantum effects; Optical properties

1. Introduction

Considerable effort has been carried out to investigate porous silicon (Si) materials because of the efficient visible photoluminescence (PL) that can be achieved from high porosity Si at room temperature (see, for example, Ref. [1]). However, the mechanism of this visible emission from porous Si is still unclear. The PL from porous Si is located at the red wavelength region and is drastically degraded by heating and by laser illumination as a result of hydrogen (H) escaping from the sample. Several researchers have reported that post treatments of porous Si such as oxidation could shift the PL spectra to the blue wavelength region [2,3]. From this blue shift the origin of the visible luminescence from porous Si is claimed to be caused by a quantum size effect. Most recently a similarity of the blue luminescence from the oxidized porous Si to a defect-related PL band at the blue to red wavelength region observed from silicon dioxide (SiO_2) was also pointed out [4,5]. Neither of these models has been conclusively proven or refuted to date.

Further, to fabricate Si crystallites with grain diameters of less than 5 nm which are also called quantum dots is a subject of interest. The motivation is to create new materials with size-tunable optical and electronic properties utilizing the Si quantum dots. In the nanometre range the band gap of the crystallites increases with decreasing size, and the electronic states are predicted to become discrete. As reported previ-

ously [6], nanocrystalline Si (nc-Si) thin films with a grain diameter of 3–5 nm emit violet and blue light at room temperature. These emissions were proposed to be caused by the confinement effect in the small Si nanocrystallites [7,8].

In this work we investigated the nc-Si samples by Fourier transform IR (FTIR) spectroscopy, X-ray photoelectron spectroscopy (XPS) and PL measurements as well as magnetic circular dichroism (MCD) measurements. It is demonstrated that the nc-Si samples exhibit size-dependent direct transition features. No evidence was observed for the SiO_2 -related luminescence. A model is also given to understand the optical transitions from indirect to direct in such Si quantum dots.

2. Experimental procedure

The nc-Si samples used in this study were fabricated by crystallization of amorphous Si (a-Si) thin films formed on Si or quartz substrates by rapid thermal annealing. The a-Si thin films with thicknesses of 100 nm were deposited on the substrates by electron beam evaporation. The thermal annealing procedure took place in a quartz tube IR furnace flushed with nitrogen over the temperature range from 1023 K to 1223 K at annealing times from 10 s to 2 h. Hydrogen (H) contamination was carefully avoided during the process. Structural investigations were carried out by X-ray diffraction (XRD) and transmission electron mi-

croscopy (TEM) to determine the average grain diameter [7]. By controlling the annealing temperature and time a series of nc-Si thin films with average grain diameters from 3 to 10 nm were formed. After crystallization the nc-Si samples were separated into several parts for PL, FTIR and XPS measurements. The FTIR measurements were carried out by using a surface reflection mode in order to remove the influence of the substrates. For the XPS measurements, the samples were slightly etched by an argon ion beam to obtain depth distributions of the signals. PL measurements were performed before and after the oxidation of the nc-Si samples at the same measurement points using the 325 nm line of an He-Cd laser excitation. The luminescence was analysed by a 250 mm grating monochromator and detected by a photomultiplier tube using photon counting detection. Oxidation of the nc-Si samples was performed in dry oxygen (O₂) atmosphere at 1273 K. For MCD measurement performed at 4.8 K, the right and left circularly polarized and modulated probe beams from a tungsten lamp were focused on the nc-Si film mounted at the centre of a superconducting magnet. The modulated signals were detected by a Si photodiode using lock-in detection. The applied magnetic field was 0, 2, 4 and 6 T. The measurements were also performed with a reversed magnetic field at 6 T to eliminate background signals.

3. Results and discussions

Shown in Fig. 1 is a cross-sectional TEM photograph

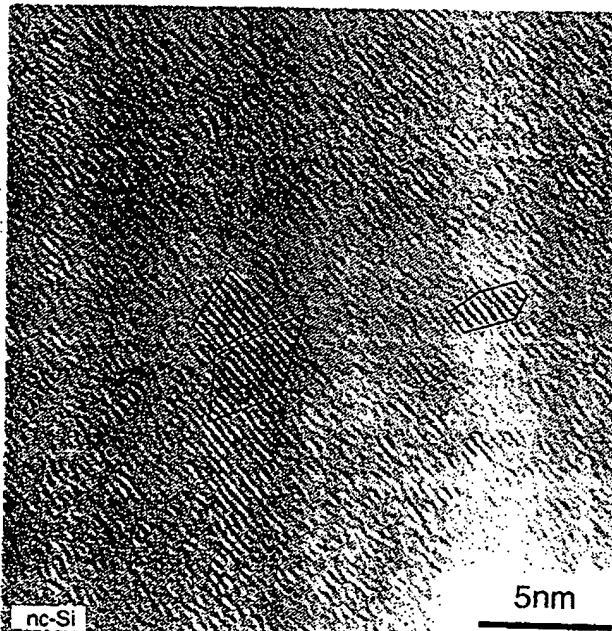


Fig. 1. Cross-sectional TEM photograph of an nc-Si thin film. Most of the crystallites grow in the <111> direction.

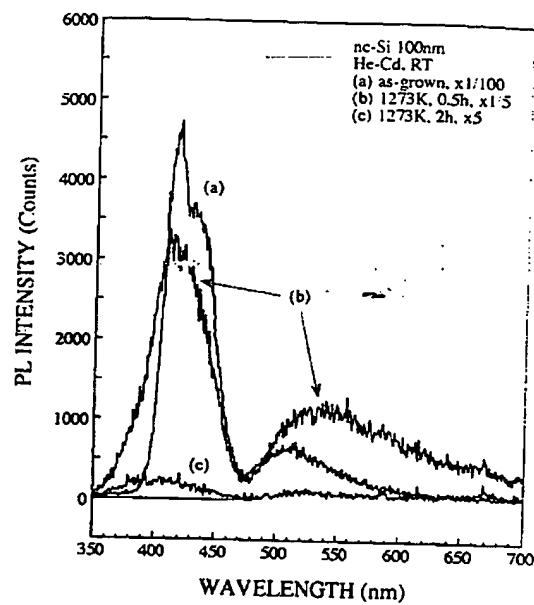


Fig. 2. PL spectra from as-grown nc-Si thin film (spectrum a) and nc-Si thin films oxidized for 0.5 h (spectrum b) and 2 h (spectrum c) at room temperature.

of an nc-Si sample having an average grain size of 3 nm. TEM observations indicate that the nanocrystalline phase achieved here in nc-Si samples consists of a random grain size distribution from 2 to 5 nm at its surface. The PL spectra also change in different areas at the surface. XRD measurements reveal a distribution of small crystallites having different crystal growth directions. More than 90% of the crystallites have <111> and <220> crystal growth directions. The grain sizes achieved here depend on the crystallization conditions. At the interface between the nc-Si layer and the substrate sharp needle-like Si crystallites grown in the <111> direction with length ranging from 10 to 50 nm are observed [7]. In the regions near the surface rectangularly shaped crystallites with sharp grain boundaries occur. The length of the crystallites in the surface region varies from 2 to 5 nm in the <111> direction. The width was estimated to vary from 1 to 3 nm. A strain-related crystallization process in the original a-Si layer might lead to such small sizes and sharp boundaries of the nanocrystallites. The periodic spacing of the lattice of the nanocrystallites is 0.31 nm corresponding to that of the {111} planes of the Si substrate.

Curve a in Fig. 2 shows the PL spectrum of an as-crystallized nc-Si thin film at room temperature under He-Cd continuous-wave laser excitation. The PL spectra exhibit a violet and blue luminescence band including separated peaks at the wavelength region from 380 to 470 nm as well as a defect-like luminescence band at longer wavelengths (470-620 nm). Numbers and positions of the peaks depend on preparation conditions. The emissions are intense and exhibit no

intensity degradation during laser illumination over the temperature range from 4.2 K to 373 K. This fact is very different from the degradation behaviour of the PL from porous Si [1]. For PL measurements using pulsed nitrogen laser excitation with a pulse width of 270 ps only the violet and blue emissions at 380–470 nm can be observed. The emissions at longer wavelengths disappear. These emissions might be caused by defect levels such as dangling bonds and the surrounding a-Si phase at the surface of the nanocrystallites because their relative intensities are weaker in samples having less a-Si phase.

After the PL measurements of Fig. 2, curve a, we oxidized the sample six times at 1273 K for 0.5 h. For every oxidation the PL was measured at the same point using the same PL system. The sample oxidized once (0.5 h) also exhibits blue emission with intensities one order of magnitude weaker than that of spectrum a. However, the separated peaks disappeared in the spectrum and a broader peak is observed, as shown in Fig. 2 by spectrum b. The emission at 470–620 nm also becomes weaker and broader. The intensities of the blue emission decrease drastically with further oxidation. Curve c in Fig. 2 is the spectrum measured after oxidation four times (2 h). The luminescence becomes very weak and is comparable with the background level of the system. The sample oxidized six times in which only SiO_2 exists shows no detectable luminescence. As reported previously, temperature-dependent PL measurements of the nc-Si samples indicated a similar temperature coefficient of the PL peaks to that of the band gap of bulk Si [9]. The emission peaks shift to higher energies with decreasing temperature and do not change their energy separations at different temperatures. The intensities and line widths of the peaks remain constant too. In addition, the intensities of the four peaks depend linearly on the excitation power density. These facts suggest that the observed blue emissions do not originate from SiO_2 but form direct transitions in confined Si crystallites which are not affected by phonons.

In order to investigate the electron states on the surface of the nanocrystallites in the nc-Si thin film, FTIR measurements were carried out by using a reflection measurement mode to remove the influence of the substrate. Fig. 3 shows the FTIR spectra at room temperature. Curve a in Fig. 3 is a transmittance spectrum of an as-formed a-Si thin film. The absorptions of the Si–Si stretching modes (621 cm^{-1} and 1107 cm^{-1}) are very weak. After crystallization the sample exhibits strong Si–Si absorption peaks in contrast to a-Si, as shown by spectrum b in Fig. 3. It should be pointed out that in spectra a and b no H- or O-related absorption exists. In hydrogenated a-Si or in porous Si stretching vibration modes of $\text{Si}-\text{H}_x$ (670 cm^{-1} and 2100 cm^{-1}) can be detected. In nc-Si oxidized 6 times or in oxidized

Si the Si–O₂ absorption at 980 cm^{-1} – 1050 cm^{-1} exists. The FTIR spectrum of bulk Si is also shown in Fig. 3 by curve c. An identical transmittance to that of the as-formed nc-Si is obtained. These findings indicate that the nc-Si samples are not oxidized or hydrogenated and that the blue luminescence is generated by the Si nanocrystallites and not by defects or complexes related to O and H.

The XPS spectra of the Si 2p orbitals at different depths of the nc-Si thin film are shown in Fig. 4. The measurement was first carried out from the surface of the nc-Si thin film. Its spectrum, labelled "no-etching", reveals an Si–Si bond at 99.5 eV as well as a suboxide peak at 103.5 eV. The suboxide is due to natural oxidation of the nc-Si sample and consists of Si^{3+} and Si^{4+} bonding states, as shown in Fig. 4. The thickness of the natural oxide layer is estimated to be 1–1.5 nm. Subsequently, the sample was etched by an argon ion gun with an etching rate of about 5 nm min^{-1} . After 0.5 min of etching and more the XPS spectra exhibit no Si–O bonding state other than the Si–Si signal. If one considers that the blue PL of the nc-Si thin film arises from the whole layer, the possibility of SiO_2 -originated emission is eliminated by this investigation. The blue emissions from nc-Si have a very short decay lifetime (about 150 ps) and exhibit no intensity degradation [9]. In contrast, the reported blue PL from SiO_2 displays longer lifetimes ranging from the micro- to nanosecond region [4]. The most reasonable explanation is that the

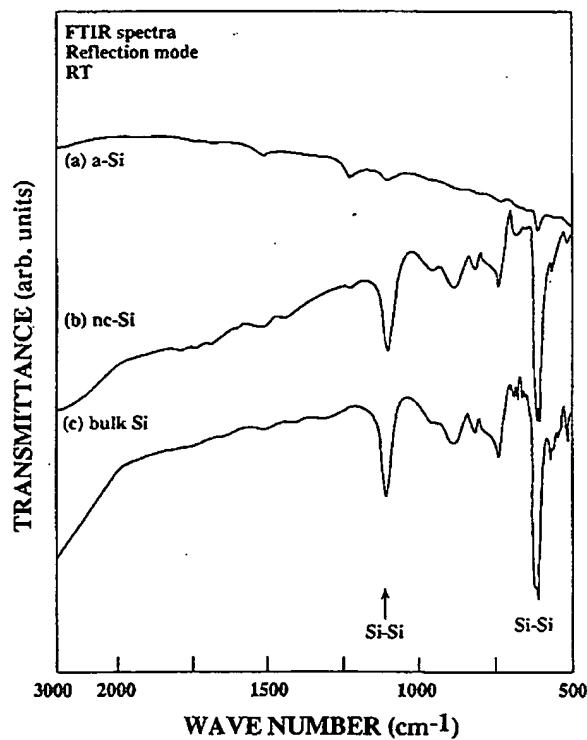


Fig. 3. FTIR spectra of a-Si (spectrum a), nc-Si (spectrum b) and bulk Si (spectrum c) samples.

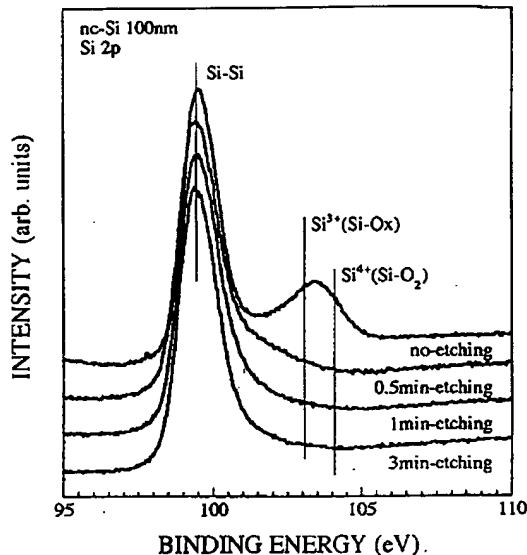


Fig. 4. XPS spectra of Si 2p orbitals at different depths in an nc-Si thin film.

blue emission from nc-Si is caused by direct transitions in the formed Si nanocrystallites which are enhanced by the quantum confinement.

MCD spectra for nc-Si samples with grain diameters of 3 nm, 6 nm and 8 nm are shown in Fig. 5. The spectrum for 8 nm indicates a negative absorbance change peak at 2.6 eV and a positive peak at 3.0 eV. The intensity of the peaks increases with increasing magnetic field. This feature is caused by the Zeeman splitting of a quantum state at 2.8 eV created by confinement of electrons in Si nanocrystallites. The negative peak shifts to higher energies corresponding to a decrease in the grain diameter indicating the quantum size effect. The quantum states at 2.95 eV and 3.1 eV

created by the direct transitions are the reason for the negative peaks at 2.7 eV and -2.8 eV for grain sizes of 6 nm and 3 nm respectively. The estimated energy position coincides with the room temperature PL at about 3 eV (410 nm in Fig. 2) for the sample with 3 nm grain diameter. Their short lifetime supports the interpretation that the states at 3.1 eV in Fig. 5 and the PL at 3 eV originate from the direct transitions.

As shown by TEM investigations the grain size at the surface of the nc-Si thin films ranges from 2 to 5 nm. It is expected that quantum confinement effects occur in small crystallites such as these. The geometrical restriction of electron-hole pairs should lead to a strong enhancement effect of the oscillator strength of the confined levels resulting in direct transitions in zero-dimensional systems. From the reported theoretical calculations [10,11], the recombination time of transitions with an energy of about 3 eV ranges from milli- to microseconds, but is not predicted to be in the pico- to nanosecond region. It has to be pointed out, however, that all the proposed models for the quantum confinement effects in Si quantum dots overestimate the indirect nature of the optical transitions. The continuous phonon modes were used as in bulk Si to calculate the radiative transition rate, and the confinement effect on phonons which means a discrete phonon distribution in k space is neglected. No valid models are proposed for treating the direct transitions in Si quantum dots even though several reports exist about the nanosecond PL decays observed in oxide-capped Si crystallites [12]. To consider the formation of quantum states in k space for an Si dot, all the zero-phonon transitions give energies of about 3 eV. We have theoretically calculated the electron states of Si spherical dots with diameters less than 5 nm. The generated quantum states of a 2.72 nm Si spherical dot, for example, are shown in Fig. 6. Ten discrete states ($k = k_1 - k_{10}$) are formed from Γ to X in k space. The energies of the direct transitions range from 3 to 4 eV. The nature of excited electrons that relaxed from $k = k_1$ to $k = k_2$ should be different from that in the bulk owing to the confinement of phonons. This confinement leads to an enhancement effect on the probability of the direct optical transitions in such confined systems. In contrast, the probability of indirect transitions, for instance the transitions from k_u upper state to k_m lower state ($n, m = 1 - 10$) which need assistance from the confined phonons, should be decreased. The direct transitions might have a higher recombination rate than that of the indirect transitions owing to the enhancement effect of the transition probabilities of the quantum states. Thus the fast decays of the blue PL are concluded to be caused by the direct transitions in Si quantum dots. This model gives a more acceptable interpretation of the efficient blue light emission from Si nanostructures. A detailed description of the model will be published elsewhere.

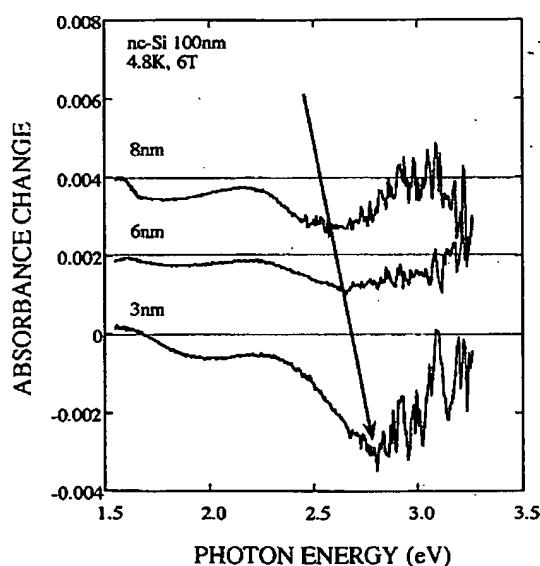


Fig. 5. MCD spectra of three nc-Si thin films.

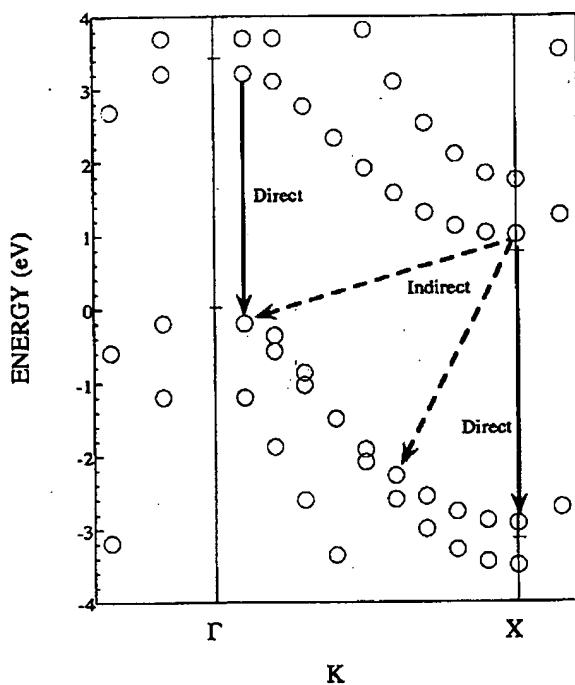


Fig. 6. Band diagram of a 2.72 nm spherical Si quantum dot. No state is formed at the Γ point.

4. Conclusion

We investigated the violet and blue light-emitting nanocrystalline Si thin films with grain diameters of 3–5 nm by FTIR spectroscopy, XPS and PL measurements as well as MCD measurements. It is demonstrated that the nc-Si samples exhibit size-dependent direct transition features. No evidence of the SiO_2 -originated luminescence was confirmed. A theoretical analysis taking the confined phonons in the Si quantum dots into account is also proposed to understand the

optical transitions from indirect to direct in such Si quantum dots. The summary of the results reported here supports the explanation of the light emission from confined levels in Si nanocrystallites. These results give a new view of the three-dimensionally confined electrons in Si-based materials.

Acknowledgments

We would like to thank Dr. S. Nagai of Komatsu Electronic Metals Co. Ltd., for realizing the TEM measurements. We would like to thank Dr. H. Isshiki and Dr. J. Kusano of RIKEN for many stimulating discussions.

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MATERIALS SCIENCE & ENGINEERING

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Solid-State Materials for Advanced Technology

Volume B35 (1995)

Proceedings of The First International Conference
on Low Dimensional Structures and Devices, 8-10 May
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0921-5093/95 \$9.50

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